Facile Preparation of 2-Imidazolines from Aldehydes with *tert*-Butyl Hypochlorite

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Received 9 April 2007

Abstract: An efficient and high-yield preparation of 2-imidazolines was achieved from aldehydes and ethylenediamines in the presence of *tert*-butyl hypochlorite. By this method, 1,3-bis(imidazolin-2-yl)benzene and 2,6-bis(imidazolin-2-yl)pyridine, which act as chiral ligands, could be prepared directly from the corresponding dialdehydes in high yields.

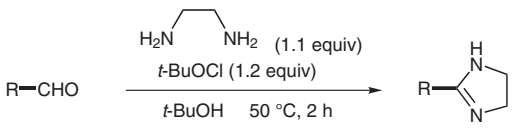
Key words: *tert*-butyl hypochlorite, 2-imidazoline, aldehydes, ethylenediamine

The synthetic study of 2-imidazoline units is very important because of their potent biological activity[1] and synthetic utility as chiral ligands.[2] To date, there are several synthetic methods for the preparation of 2-imidazolines, mainly starting from nitriles and esters.[3] Recently, an efficient one-pot preparation of 2-imidazolines from aldehydes and ethylenediamine with *N*-bromosuccinimide was reported.[4] We also reported an efficient method for the preparation of 2-imidazolines from aldehydes and molecular iodine.[5] However, in the method using molecular iodine, the yield of 2-alkylimidazolines with aliphatic aldehydes was rather low, although that of 2-arylimidazolines with aromatic aldehydes was high. Here, as part of our basic study of molecular iodine and related reagents in organic synthesis,[6] we wish to report another efficient and practical oxidative conversion of aldehydes together with ethylenediamine and *tert*-butyl hypochlorite into 2-imidazolines.

tert-Butyl hypochlorite is an inexpensive and versatile reagent, and has been applied to oxidative transformations such as the oxidation of alcohols to ketones,[7] aldehydes to acid chlorides,[8] sulfides to sulfoxides,[9] and hydroxylamines to nitroso compounds.[10] Furthermore, *tert*-butyl hypoiodite is an easily available reagent, which can be prepared from *tert*-butyl hypochlorite and metal iodide or iodine,[11] and has recently been developed for the synthesis of N-heterocycles[12] or iodocyclization.[13] Thus, the addition of *tert*-butyl hypochlorite to a mixture of benzaldehyde and ethylenediamine in *tert*-butyl alcohol provides the corresponding 2-phenylimidazoline quantitatively (Table 1, entry 1).

Under the same conditions, various aromatic and aliphatic aldehydes were treated with ethylenediamine and *tert*-butyl hypochlorite in *tert*-butyl alcohol, and the corresponding 2-aryl- and 2-alkylimidazolines were obtained in good yields (Table 1). Electron-rich aromatic aldehydes such as *p*-methoxybenzaldehyde, thiophenecarboxyaldehyde, and 1-naphthaldehyde require long reaction times (Table 1, entries 3, 4, 10, 12, and 13). When potassium iodide was added to the reaction mixture, the yield was improved, because of the formation of *tert*-butyl hypoiodite, which was

Table 1 Preparation of 2-Imidazolines in the Presence of *tert*-Butyl Hypochlorite



Entry	R	Yield[a] (%)
1	Ph	100
2	4-tolyl	98
3[b]	PMP	81
4[b,c]	PMP	99
5	4-BrC_6H_4	100
6	4-$O_2NC_6H_4$	100
7	4-NCC_6H_4	88
8	2-ClC_6H_4	99
9	2-thienyl	64
10[b]	2-thienyl	91
11	2-pyridyl	100
12[b]	1-naphthyl	81
13[b,c]	1-naphthyl	100
14[b,c]	1-adamantyl	99
15	cyclohexyl	79
16	$(CH_2)_2Ph$	98
17[b]	$(CH_2)_6Me$	95
18[b]	CHMePh	80

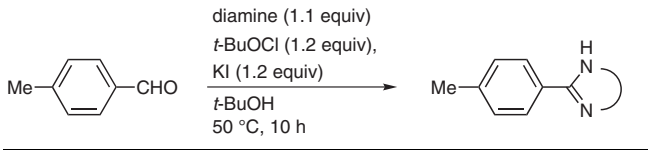
[a] Isolated yield.
[b] Reaction time was 10 h.
[c] KI (1.2 equiv) was added.

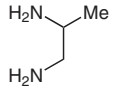
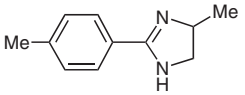
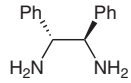
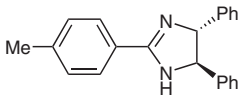
SYNTHESIS 2007, No. 13, pp 1939–1942
Advanced online publication: 18.06.2007
DOI: 10.1055/s-2007-983726; Art ID: F06607SS

generated from *tert*-butyl hypochlorite and potassium iodide in situ (Table 1, entries 4, 13, and 14). 2-Alkylimidazolines were obtained from the corresponding aldehydes in much high yields (Table 1, entries 15–18) by this method than by the molecular iodine method.

When 1-methylethylene-1,2-diamine, instead of ethylenediamine, was also treated with *p*-tolualdehyde and *tert*-butyl hypochlorite in the presence of potassium iodide, the corresponding 2-(*p*-tolyl)imidazoline was formed in good yield (Table 2, entry 1). The same treatment of *p*-tolualdehyde with (*R*,*R*)-(+)-diphenylethylenediamine provided the corresponding (*R*,*R*)-4,5-diphenyl-2-(*p*-tolyl)imidazoline in good yield (Table 2, entry 2).

Table 2 Use of Other Diamines for the Preparation of 2-Imidazolines

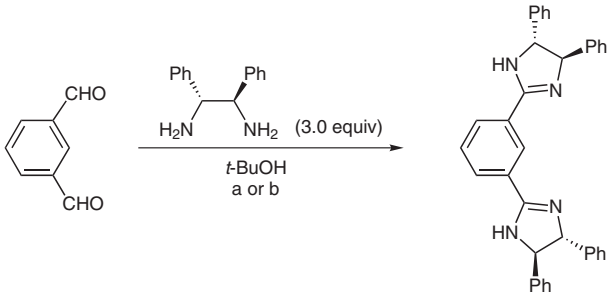


Entry	Diamine	Product	Yield[a] (%)
1			100
2			97

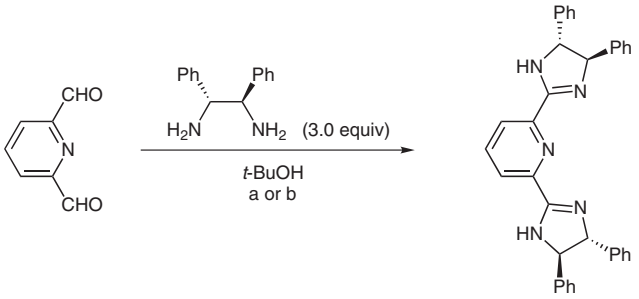
[a] Isolated yield.

Then, benzene-1,3-dicarbaldehyde (Scheme 1) and pyridine-2,6-dicarbaldehyde (Scheme 2) were treated with (*R*,*R*)-(+)-diphenylethylenediamine and *tert*-butyl hypochlorite in the presence of potassium iodide under the same conditions; this provided the corresponding 1,3-bis(imidazolin-2-yl)benzene and 2,6-bis(imidazolin-2-yl)pyridine (pybim), which are known as chiral ligands, in good yields. When the reactivities of the molecular iodine method and the *tert*-butyl hypochlorite method for the formation of pybim are compared, the former method is better than the latter. However, *tert*-butyl hypochlorite can be used for both aromatic aldehydes and aliphatic aldehydes to provide the corresponding 2-substituted imidazolines in good yields. Moreover, pybim was recently prepared from 2,6-dicyanopyridine in two steps,[14] whereas it could be obtained in high yield in a single step from the commercially available pyridine-2,6-dicarbaldehyde by the *tert*-butyl hypochlorite method presented here.

In summary, various 2-aryl- and 2-alkylimidazolines could be obtained easily and in good yields by the reaction between aldehydes and ethylenediamine in the presence of *tert*-butyl hypochlorite. Moreover, 1,3-bis(imidazolin-2-yl)benzene and 2,6-bis(imidazolin-2-yl)pyridine, a chiral ligand, could be directly prepared from the corresponding dialdehydes in high yields. Further synthetic



Scheme 1 *Reagents and conditions*: (a) I_2 (3.5 equiv), K_2CO_3 (4.0 equiv), 70 °C, 3 h (98%); (b) *t*-BuOCl (3.5 equiv), KI (3.5 equiv), 50 °C, 10 h (100%).



Scheme 2 *Reagents and conditions*: (a) I_2 (3.5 equiv), K_2CO_3 (4.0 equiv), 70 °C, 3 h (92%); (b) *t*-BuOCl (3.5 equiv), KI (3.5 equiv), 50 °C, 10 h (76%).

study of the present reactions is under way in this laboratory.

Melting points were determined on a Yamato MP-21 apparatus and are uncorrected. IR spectra were recorded on a JASCO FT/IR-200 infrared spectrometer of samples prepared as KBr pellets. NMR spectra were recorded of samples in $CDCl_3$ (unless stated otherwise) on a JEOL JNM-LA-400 spectrometer. HRMS–FAB determinations were carried out on a JEOL JMS-AX500 spectrometer, and HRMS measurements were obtained on a JEOL HX-110 instrument.

2-Imidazolines from Aldehydes and Ethylenediamines in the Presence of *tert*-Butyl Hypochlorite; Typical Procedure

To a soln of PhCHO (106.0 mg, 1 mmol) in *t*-BuOH (10 mL) was added ethylenediamine (66.1 mg, 1.1 mmol). The mixture was stirred at r.t. under an argon atmosphere for 30 min, and then *t*-BuOCl (130.3 mg, 1.2 mmol) was added, and the mixture was stirred at 50 °C. After 2 h, the mixture was quenched with sat. aq Na_2SO_3 (10 mL) and was extracted with $CHCl_3$ (3 × 10 mL). The organic layer was washed with sat. aq K_2CO_3 (1 × 10 mL) and brine (1 × 20 mL), and dried (Na_2SO_4). After filtration, the mixture was evaporated; this provided almost pure 2-(phenyl)imidazoline; yield: 100% yield. If necessary, the product can be purified by flash column chromatography (neutral silica gel, $CHCl_3$–Et_3N, 10:1).

2-(Phenyl)imidazoline

Mp 101.5–102 °C (Lit.[15] 100–101 °C).

IR (KBr): 3200, 2930, 1600, 1510, 1270, 985, 695 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 3.90 (s, 4 H), 7.46 (t, *J* = 7.2 Hz, 2 H), 7.57 (t, *J* = 7.2 Hz, 1 H), 7.95 (d, *J* = 7.2 Hz, 2 H).

2-(4-Tolyl)imidazoline
Mp 181–182 °C (Lit.[15] 181 °C).

IR (KBr): 3140, 2925, 1600, 1495, 985, 830 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 2.38 (s, 3 H), 3.77 (s, 4 H), 7.21 (d, *J* = 8.3 Hz, 2 H), 7.67 (d, *J* = 8.3 Hz, 2 H).

2-(4-Methoxyphenyl)imidazoline
Mp 136–138 °C (Lit.[16] 137–139 °C).

IR (KBr): 3120, 2830, 1605, 1490, 1255, 1035, 845 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 3.77 (s, 4 H), 3.84 (s, 3 H), 6.91 (d, *J* = 8.9 Hz, 2 H), 7.73 (d, *J* = 8.9 Hz, 2 H).

2-(4-Bromophenyl)imidazoline
Mp 177–177.5 °C.

IR (KBr): 3150, 2930, 1610, 1470, 1270, 1010, 835 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 3.79 (s, 4 H), 7.54 (d, *J* = 8.7 Hz, 2 H), 7.65 (d, *J* = 8.7 Hz, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 125.0, 128.5, 129.4, 131.6, 163.8.

HRMS–FAB: *m*/*z* $[M + H]^+$ calcd for $C_9H_{10}N_2Br$: 225.0027; found: 225.0021.

2-(4-Nitrophenyl)imidazoline
Mp 235–237 °C (Lit.[17] 231 °C).

IR (KBr): 3180, 2935, 1580, 1520, 1335, 1105, 855 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 3.85 (s, 4 H), 7.95 (d, *J* = 8.9 Hz, 2 H), 8.27 (d, *J* = 8.9 Hz, 2 H).

2-(4-Cyanophenyl)imidazoline
Mp 195–196 °C.

IR (KBr): 3160, 2230, 1595, 1490, 1275, 985, 850 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 3.83 (s, 4 H), 7.70 (d, *J* = 8.5 Hz, 2 H), 7.88 (d, *J* = 8.5 Hz, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 114.2, 118.4, 127.7, 132.4, 134.7, 163.2.

HRMS–FAB: *m*/*z* $[M + H]^+$ calcd for $C_{10}H_{10}N_3$: 172.0875; found: 172.0883.

2-(2-Chlorophenyl)imidazoline
Mp 83 °C (Lit.[18] 69–70 °C).

IR (KBr): 3100, 2920, 1610, 1505, 1260, 985, 765 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 3.80 (s, 4 H), 7.28–7.41 (m, 3 H), 7.78 (dd, *J* = 7.5 and 1.9 Hz, 1 H).

2-(2-Thienyl)imidazoline
Mp 175 °C (Lit.[16] 175–177 °C).

IR (KBr): 3150, 2935, 1595, 1495, 1270, 985, 710 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 3.78 (s, 4 H), 7.06 (dd, *J* = 4.9, 3.7 Hz, 1 H), 7.36 (dd, *J* = 3.7, 0.9 Hz, 1 H), 7.40 (dd, *J* = 4.9, 0.9 Hz, 1 H).

2-(2-Pyridyl)imidazoline
Mp 95–96 °C.

IR (KBr): 3270, 1595, 1505, 1280, 975, 805, 750 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 3.85 (s, 4 H), 7.36 (dd, *J* = 4.8, 1.2 Hz, 1 H), 7.77 (td, *J* = 7.8, 0.7 Hz, 1 H), 8.14 (d, *J* = 7.8 Hz, 1 H), 8.57 (dt, *J* = 4.8, 0.7 Hz, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 50.4, 122.2, 125.0, 136.5, 148.5, 148.6, 164.2.

HRMS–FAB: *m*/*z* $[M + H]^+$ calcd for $C_8H_{10}N_3$: 148.0875; found: 148.0876.

2-(1-Naphthyl)imidazoline
Mp 131–133 °C (Lit.[19] 134 °C).

IR (KBr): 3140, 2860, 1570, 1515, 1270, 980, 775 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 3.89 (s, 4 H), 7.45–7.57 (m, 3 H), 7.75 (dd, *J* = 7.1, 1.2 Hz, 1 H), 7.85–7.91 (m, 2 H), 8.68 (d, *J* = 8.5 Hz, 1 H).

2-(1-Adamantyl)imidazoline
Mp 162.5–163.5 °C.

IR (KBr): 3230, 3000, 1590, 1495, 1250, 1085, 980 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 1.69–1.77 (m, 6 H), 1.86–1.87 (m, 6 H), 2.03 (m, 3 H), 3.56 (s, 4 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 28.1, 35.1, 36.5, 40.3, 49.4, 174.5.

HRMS–FAB: *m*/*z* $[M + H]^+$ calcd for $C_{13}H_{21}N_2$: 205.1705; found: 205.1703.

2-Cyclohexylimidazoline
Mp 130.5–131 °C (Lit.[15] 134 °C).

IR (paraffin): 3085, 1600, 1510, 1275, 1060, 980 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 1.16–1.43 (m, 5 H), 1.67–1.92 (m, 5 H), 2.22 (tt, *J* = 11.5, 3.4 Hz, 1 H), 3.56 (s, 4 H).

2-Phenethylimidazoline
Mp 101–103 °C.

IR (KBr): 3160, 2925, 1605, 1500, 1285, 960, 700 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 2.54 (t, *J* = 8.0 Hz, 2 H), 2.96 (t, *J* = 8.0 Hz, 2 H), 3.55 (br s, 4 H), 7.21–7.36 (m, 5 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 31.3, 32.9, 126.3, 128.3, 128.6, 141.1, 167.2.

HRMS–FAB: *m*/*z* $[M + H]^+$ calcd for $C_{11}H_{15}N_2$: 175.1235; found: 175.1237.

4-Methyl-2-(4-tolyl)imidazoline
Mp 162–164 °C.

IR (paraffin): 3400, 1590, 1540, 1330, 1015, 830, 730 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 1.29 (d, *J* = 6.3 Hz, 3 H), 2.38 (s, 3 H), 3.36 (br s, 1 H), 3.91 (br s, 1 H), 4.11 (br s, 1 H), 7.20 (d, *J* = 8.0 Hz, 2 H), 7.66 (d, *J* = 8.0 Hz, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.3, 22.0, 51.2, 53.2, 118.3, 129.7, 130.0, 146.4, 164.1.

HRMS–FAB: *m*/*z* $[M + H]^+$ calcd for $C_{11}H_{15}N_2$: 175.1235; found: 175.1236.

(4*R*,5*R*)-4,5-Diphenyl-2-(4-tolyl)imidazoline
Mp 146.5 °C.

IR (paraffin): 3150, 1600, 1130, 1020, 830, 765, 700 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 2.43 (s, 3 H), 4.75 (br s, 1 H), 5.07 (br s, 1 H), 5.33 (br s, 1 H), 7.26–7.37 (m, 12 H), 7.84 (d, *J* = 8.2 Hz, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.4, 126.7, 127.3, 127.4, 128.6, 129.2, 143.6, 163.0.

HRMS–FAB: *m*/*z* $[M + H]^+$ calcd for $C_{22}H_{21}N_2$: 313.1705; found: 313.1676.

1,3-Bis[(4*R*,5*R*)-4,5-diphenyl-2-imidazolin-2-yl]benzene
Mp 136.5–138 °C.

IR (neat): 3062, 1570, 1493, 1452, 1270, 754, 696 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 4.93 (br s, 4 H), 7.28–7.37 (m, 20 H), 7.57 (t, *J* = 7.8 Hz, 1 H), 8.11 (d, *J* = 7.8 Hz, 2 H), 8.50 (s, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 126.1, 126.6, 127.6, 128.7, 128.9, 129.0, 130.4, 143.1, 162.4.

HRMS–FAB: *m*/*z* $[M + H]^+$ calcd for $C_{36}H_{31}N_4$: 519.2549; found: 519.2513.

2,6-Bis[(4*R*,5*R*)-4,5-diphenyl-2-imidazolin-2-yl]pyridine
Mp 124–125 °C (Lit.[14] 123–126 °C).

IR (neat): 3028, 1606, 1564, 1452, 999, 752, 698 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 4.79 (d, *J* = 8.7 Hz, 2 H), 5.20 (d, *J* = 8.7 Hz, 2 H), 6.42 (br s, 2 H), 7.25–7.35 (m, 20 H), 7.98 (t, *J* = 7.8 Hz, 1 H), 8.48 (d, *J* = 7.8 Hz, 2 H).

Acknowledgment

Financial support from a Grant-in-Aid for Scientific Research (No. 16655012) from the Ministry of Education, Science, Sports, and Culture of Japan is gratefully acknowledged.

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